

TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 USC § 371

AC08 Rec'd PCT/E10 05 FEB 2001
84-472

US APPLICATION NO. (If known, see 37 CFR 1.53)

09/762211

INTERNATIONAL APPLICATION NO. □

PCT/N099/00287

INTERNATIONAL FILING DATE

17 September 1999

PRIORITY DATE CLAIMED

25 September 1998

TITLE OF INVENTION

CARBON MEDIA FOR STORAGE OF HYDROGEN

APPLICANT(S) FOR DO/EO/US

KVAERNER TECHNOLOGY AND RESEARCH LIMITED

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

- 1 ☒ This is a **FIRST** submission of items concerning a filing under 35 USC § 371 □
- 2 ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 USC § 371 □
- 3 ☐ This is an express request to begin national examination procedures (35 USC § 371(f)) □ The submission must include items (5), (6), (9) and (21) indicated below □
- 4 ☐ The US has been elected by the expiration of 19 months from the priority date (Article 31) □
- 5 ☒ A copy of the International Application as filed (35 USC § 371(c)(2))
- a ☐ is attached hereto (required only if not communicated by the International Bureau) □
- b ☒ has been communicated by the International Bureau □
- c ☐ is not required, as the application was filed in the United States Receiving Office (RO/US) □
- 6 ☐ An English language translation of the International Application as filed (35 USC § 371(c)(2)) □
- a ☐ is attached hereto □
- b ☐ has been previously submitted under 35 USC § 54(d)(4) □
- 7 ☐ Amendments to the claims of the International Application under PCT Article 19 (35 USC § 371(c)(3))
- a ☐ are attached hereto (required only if not communicated by the International Bureau) □
- b ☐ have been communicated by the International Bureau □
- c ☐ have not been made; however, the time limit for making such amendments has NOT expired □
- d ☐ have not been made and will not be made □
- 8 ☐ An English language translation of the amendments to the claims under PCT Article 19 (35 USC § 371(c)(3)) □
- 9 ☐ An oath or declaration of the inventor(s) (35 USC § 371(c)(4)) □
- 10 ☒ An English language translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 USC § 371(c)(5)) □

Items 11 to 20 below concern document(s) or information included:

- 11 ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98 □
- 12 ☐ An assignment document for recording □ A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included □
- 13 ☐ A **FIRST** preliminary amendment □
- 14 ☐ A **SECOND** or **SUBSEQUENT** preliminary amendment □
- 15 ☐ A substitute specification □
- 16 ☐ A change of power of attorney and/or address letter □
- 17 ☐ A computer-readable form of the sequence listing in accordance with PCT Rule 13ter(2) and 35 USC § 54(d)(4) □
- 18 ☐ A second copy of the published international application under 35 USC § 54(d)(4) □
- 19 ☐ A second copy of the English language translation of the international application under 35 USC § 54(d)(4) □
- 20 ☐ Other items or information:

21 ☐ The following fees are submitted:

09/762211

CALCULATIONS PTO USE ONLY

BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)):

JC02 Rec'd

PCT/PTO 05 FEB 2001

Neither international preliminary examination fee (37 CFR 1.482)
nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO
and International Search Report not prepared by the EPO or JPO ☐ \$1000.00

International preliminary examination fee (37 CFR 1.482) not paid to
USPTO but International Search Report prepared by the EPO or JPO ☐ \$860.00

International preliminary examination fee (37 CFR 1.482) not paid to USPTO
but international search fee (37 CFR 1.445(a)(2)) paid to USPTO ☐ \$710.00

International preliminary examination fee (37 CFR 1.482) paid to USPTO
but all claims did not satisfy provisions of PCT Article 33(1)-(4) ☐ \$690.00

International preliminary examination fee (37 CFR 1.482) paid to USPTO
and all claims satisfied provisions of PCT Article 33(1)-(4) ☐ \$100.00

ENTER APPROPRIATE BASIC FEE AMOUNT =

\$ 860.00

Surcharge of \$130.00 for furnishing the oath or declaration later than ☐ 20 ☒ 30
months from the earliest claimed priority date (37 CFR 1.492(e)) ☐

\$ 130.00

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	\$
Total claims	11 - 20 =	0	x \$18.00	\$
Independent claims	3 - 3 =	0	x \$80.00	\$
MULTIPLE DEPENDENT CLAIM(S) (if applicable) 4			+ \$270.00	\$ 270.00

TOTAL OF ABOVE CALCULATIONS =

\$

☐ Applicant claims small entity status ☐ See 37 CFR 1.27 ☐ The fees indicated above
are reduced by 1/2 ☐

\$

SUBTOTAL =

\$ 1,260.00

Processing fee of \$130.00 for furnishing the English translation later than ☐ 20 ☐ 30
months from the earliest claimed priority date (37 CFR 1.492(f)) ☐

\$

TOTAL NATIONAL FEE =

\$ 1,260.00

Fee for recording the enclosed assignment (37 CFR 1.21(h)) ☐ The assignment must be
accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31) ☐ \$40.00 per property +

\$

TOTAL FEES ENCLOSED =

\$ 1,260.00

Amount to be
refunded:

\$

charged:

\$

a ☒ A check in the amount of \$ 1,260.00 to cover the above fees is enclosed ☐

b ☐ Please charge my Deposit Account No. _____ in the amount of \$ _____ to cover the above fees ☐
A duplicate copy of this sheet is enclosed ☐

c ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any
overpayment to Deposit Account No. 500687 ☐ A duplicate copy of this sheet is enclosed ☐

d ☐ Fees are to be charged to a credit card ☐ **WARNING:** Information on this form may become public ☐ **Credit card
information should not be included on this form** ☐ Provide credit card information and authorization on PTO-2038 ☐

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR
1.37 (a) or (b)) must be filed and granted to restore the application to pending status ☐

SEND ALL CORRESPONDENCE TO:

W. Warren Taltavull
Manelli, Denison & Selter PLLC
2000 M Street N.W.
Washington, DC 20036
Suite 700

SIGNATURE

W. Warren Taltavull
NAME

25647
REGISTRATION NUMBER

09762211

CARBON MEDIA FOR STORAGE OF HYDROGEN

5 FIELD OF INVENTION

This invention relates to a carbon media for storage of hydrogen.

10 BACKGROUND OF THE INVENTION

- There is currently an intense interest in carbon materials due to their unique and novel properties. For instance, the carbon materials may be useful to achieve high hydrogen energy storage, for use in purification processes as well as for different applications within the electronical/pharmaceutical sector. The properties are sensitive to the micro-structure of the carbon material, which can be varied by the nanostructure ordering (graphitisation level). The nanostructure ordering spans from non-crystalline qualities such as conventional carbon black (furnace black, thermal black) to crystalline qualities such as graphite and novel carbon materials with graphitic structures. The nanostructure ordering can be described in terms of the distance between the graphite layers, which will vary from 3.40 Å for a ordered crystalline structure to 3.60 Å for non-crystalline materials.
- 25 The recent interest in carbon materials for use as a storage medium has mainly been focused on novel materials with graphitic structures where the degree of graphitisation and the introduction of rings other than hexagons in the network is of vital importance. Fullerenes are examples of novel graphitic structures where the introduction of 12 pentagons in the hexagonal network results in closed shells [1]. Carbon nanotubes is also an example of such possibilities [2]. Open conical structures are yet another example of possible graphitic structures, but only three of five possible kinds have ever been synthesised [3, 4, 5].
- 35 Recent interest in fullerenes and nanotubes is amongst other connected to their use in the field of hydrogen storage. Hence, for nanotubes a hydrogen storage of amazingly 75wt% is reported [6]. If this is the case, it will

probably represent the break-through concerning a practical hydrogen storage system for use in the transportation sector. It is indicated that future fuel cell cars using this storage technology may achieve a range of about 8000 km.

- 5 In the case of fullerenes, more than 7wt% of reversibly added hydrogen is achieved [7, 8, 9]. Fullerenes has also been used in a solid phase mixture with inter-metallic compounds or metals to achieve high contents of hydrogen, i.e. 24-26 H atoms per fullerene molecule [10].
- 10 Flat graphitic material formed of stacks of two-dimensional sheets has high surface area for adsorption of guest elements and compounds. However, in such materials the adsorption process is probably limited by diffusion. The larger the graphitic domain, the slower the adsorption will be. Of potential interest would be highly graphitised materials where domains are small so
- 15 that the guest material would readily reach all the graphitic micro domains by percolation through the bulk carbon material. The accessibility to the micro-domains could be further enhanced if some or all the domains had topological disclination, preferably each domain having less or equal than 300° disclination to provide cavities, or micro-pores, for the flow of guest
- 20 material.

- A common problem with the present methods for synthesising these graphitic materials is the low production yield. The fullerenes are most often synthesised by vaporising graphite electrodes via carbon-arc discharges in a
- 25 reduced inert gas atmosphere. There has been reported a conversion rate into fullerenes of 10-15%, corresponding to a generation rate of nearly 10 grams per hour [11].

- The carbon-arc method is also the most frequently used method for
- 30 production of carbon nanotubes. Nanotubes yields of about 60% of the core material has been obtained at optimal conditions [2]. Still, the achieved production is in gram quantities.

- Small unspecified amounts of open conical carbon structures are obtained by
- 35 resistively heating a carbon foil and further condensing the carbon vapour on a highly-oriented pyrolytic graphite surface [3, 4]. The cone angles produced by this method was approximately 19° [3], and 19° as well as 60° [4].

Resistive heating of a carbon rod, with further deposition on cooler surfaces was used to produce cones with apparent cone angles of approximately 39° [5]. It can be shown from a continuous sheet of graphite that only five types of cones can be assembled, where each domain is uniquely defined by its
5 topological disclination TD given by the general formula:

$$TD = N \times 60 \text{ degrees}, \text{ where } N = 0, 1, 2, 3, 4 \text{ or } 5,$$

The structure of such graphitic domains can be grossly described as stacks of
10 graphitic sheets with flat ($N = 0$) or conical structures ($N = 1$ to 5). Hence, two of these, holding cone angles of 83.6° and 112.9°, has not been reported so far.

15 SUMMARY OF THE INVENTION

An object of this invention is to provide a carbon media for storage of hydrogen. This object is achieved by a media characterised in that it comprises known and novel crystalline or non-crystalline materials and that
20 it is produced by a two-step plasma process. By changing the process parameters of the plasma process, the nanostructure ordering of the carbon material can be varied in such a way that the desired microstructure for optimum hydrogen storage is achieved. These microstructures may either be conventional carbon black graphitic carbon black and/or novel carbon
25 materials such as cones, fullerenes or nanotubes.

In the one-step plasma process conventional carbon black or graphitic carbon black can be formed. A such process is described in for instance EP 0 636
30 162. The resulting carbon material may have a surface area (BET) of 5-250 m²/g and a dibutyl phthalate absorption (DBP) of 40-175 ml/100g.

In the two-step plasma process, a hydrocarbon feed material is sent through a plasma zone and becomes partly dehydrogenated in the first step to form polycyclic aromatic hydrocarbons (PAHs), and is then sent through second
35 plasma zone to become completely dehydrogenated to form micro-domain graphitic materials in the second step. By micro-domain graphitic materials we mean fullerenes, carbon nanotubes, open conical carbon structures (also

named micro-cones), flat graphitic sheets, or a mixture of two or all of these. The novel part of the carbon material is open carbon micro-cones with total disclination degrees 60° and/or 120° , corresponding to cone angles of respectively 112.9° and/or 83.6° .

5

Another object of this invention is to provide a carbon media for storage of hydrogen comprising known and novel micro-domain materials, characterised in that the media is produced in industrial scale with large yield rates of up to above 90% by a one or two-step plasma process.

10

The invention also relates to use of the carbon media as a storage media for hydrogen.

BRIEF DESCRIPTION OF THE FIGURES

15

FIG. 1 shows a schematic figure of the reactor and the surrounding equipment.

20

FIG. 2 shows a transmission electron microscope photograph of the samples revealing the various types of open micro-conical carbons of the invention.

25

FIG. 3 shows the projected angles for perfect graphitic cones, i.e. 19.2° , 38.9° , 60° , 83.6° and 112.9° , which represents total disclination of 300° , 240° , 180° , 120° and 60° , respectively. In addition a graphitic sheet, having a projected angle of 180° and a total disclination of 0° , is shown.

30

FIG. 4A, 4B, 4C, 4D and 4E shows example of domains for each type of disclination 60° , 120° , 180° , 240° and 300° , respectively, present in the invention.

DETAILED DESCRIPTION OF THE INVENTION

35

The hydrogen storage capacity of the media is linked to the small size of the domains and the presence of various topographies in the material. These are useful for the incorporation of guest elements and compounds such as hydrogen. Also, the space between the domains will provide micro-pores for

the flow of guest material so that it can reach each domain and the small size of the domains will allow rapid diffusion of guest material in and out of each layer composing them.

- 5 The carbon media for storage of hydrogen in the present invention consists of known and novel micro-domain materials, such as conventional carbon black, fullerenes, carbon nanotubes, open conical carbon structures (also named micro-cones), or flat graphitic sheets alone, or a mixture of two or all of these. The amount present of any of the carbon structures, conventional
- 10 carbon black, fullerenes, carbon nanotubes, micro-cones or flat graphitic sheets, in the media can be anywhere between 0 and 100 wt% based on the total mass of the hydrogen storage media. Further, all possible mixtures of these structures can be present.
- 15 The novel carbon material is open carbon micro-cones with total disclination degrees 60° and/or 120° , corresponding to cone angles of respectively 112.9° and/or 83.6° . It can be shown that if a cone is made up of an uninterrupted sheet of graphite, except at the open edge, only five types are possible due to the symmetry of graphite. These corresponds to a total disclination of 60° ,
- 20 120° , 180° , 240° and 300° . A total disclination of 0° corresponds to a flat domain. Fig. 3 shows schematically the projected angles of these structures. Examples of each of these types of domains are shown in Fig. 4A, 4B, 4C, 4D and 4E. It is important to notice that all the cones are closed in the apex. The novel carbon material of this invention comprises micro-domains of
- 25 graphite of well-defined total disclinations TD (curvature), which have discrete values given by the formula

$$TD = N \times 60 \text{ degrees} \quad , \text{ where } N = 0, 1, 2, 3, 4 \text{ or } 5,$$

- 30 and corresponds to the effective number of pentagons necessary to produce the particular total disclination.

- 35 The hydrogen storage media is produced in industrial scale by decomposition of hydrocarbons into carbon and hydrogen in a one or two-step plasma based process. The plasma arc is formed in a plasma generator which consists of tubular electrodes, wherein the inner electrode is supplied with electrical direct voltage with one polarity and wherein the external electrode is

connected to the opposite polarity from a power supply. The plasma generator is installed in connection with a decomposition reactor wherein the reactor is designed as a defined heat insulated chamber with an outlet for end products. The plasma gas is recycled from the process. Further description of the general process and the equipment is described in the applicant's European patent EP 0 636 162.

The structure of the resulting carbon material will depend on the following three process parameters: The hydrocarbon feed rate, the plasma gas enthalpy and the residence time. By varying these parameters the resulting carbon material will either be available as conventional carbon black, as micro-domain materials or a mixture of both. In the following we will describe the process parameters for optimising on micro-domain materials. This illustrates the capability of the process to produce the carbon material which is most suited as a storage media for hydrogen.

Experimental results indicates that the total disclination nearly always is determined in the nucleation stage. It is earlier found that the probability of forming pentagons in the seed depends on the temperature [12]. Hence, by varying the process parameters, including but not limited to increasing the reaction temperature, the number of pentagons in the seed may increase. This may in turn result in formation on nanotubes or closed shells.

The hydrocarbons are introduced into the decomposition reactor in the vicinity of the plasma arc zone by use of a self-invented nozzle which is aligning the hydrocarbon spray in the axial direction of the reactor.

Energy is supplied from the plasma arc to heat the plasma gas. Some of the energy from the arc will be used to heat the surrounding reactor walls as well as the plasma generator itself. The resulting energy content of the plasma gas (the plasma gas enthalpy) is sufficient to evaporate the hydrocarbons. The hydrocarbons starts a cracking and polymerisation process, which results in the formation of PAHs. The PAHs are the basis of graphitic sheets forming the micro-domains. The plasma gas enthalpy is kept at such a level that the main fraction of the gaseous hydrocarbons does not reach pyrolysis temperatures at the specified feedstock rate and residence time used. However, a small fraction of the feedstock will inevitably achieve sufficient

energy during the residence time in the reactor to reach pyrolysis temperature and is consequently converted to conventional carbon black. This fraction should be kept as low as possible.

- 5 The PAHs leaves the reactor along with the plasma gas and is once more introduced in the reactor as a part of the plasma gas. The plasma gas enters the energy intensive plasma arc zone, wherein the PAHs during a fraction of a second are converted to graphitic seeds. These seeds are dictating the shape of the micro-domains. PAHs formed when introducing fresh feedstock will
10 result in growth of the seeds to form graphitic micro-domains.

- Another alternative is to introduce the PAH containing plasma gas in a subsequent chamber or reactor which also is equipped with a plasma generator and an inlet for hydrocarbon feedstock to convert the PAHs into
15 graphitic micro-domains.

- The feedstock feed rate for optimising on graphitic micro-domain materials is in the range of 50-150 kg/h in a reactor employed by the inventor, but is not limited by this range. Both lower and higher feedstock feed rates might be
20 used. The yield of the graphitic micro-domain material is higher than 90% under optimal conditions. Taken into account the feedstock feed rate utilised, industrial amounts of micro-domain carbon material is achieved. By further up-scaling this will result in a price which is on the same level as commercial carbon black per unit weight of the material.

- 25 Fig. 1 shows a schematic drawing of the reactor. Further details concerning the reactor and the surrounding equipment is described in the applicant's European patent EP 0 636 162.

- 30 Fig. 2 shows a typical example of the content of the micro-domain material. Each piece in the sample forms a single graphitic domain and the alignment of the sheets in each domain is typically turbostratic, as determined from electron microscopy. The diameter of the domains is typically less than 5 micrometers and the thickness less than 100 nanometers.

- 35 In the following, it will be demonstrated that by altering the conditions in the plasma reactor, it is possible to produce either conventional carbon black or

micro-domain graphitic materials. According to the present invention, both may be used as a hydrogen storage media. In Example 1, the process parameters are chosen in such a way that conventional carbon black is formed at the first (and only) cycle of hydrocarbons through the reactor. By varying the feedstock feed rate, the plasma gas enthalpy and the residence time, it is shown in Example 2 that at the second cycle through the reactor, micro-domain graphitic materials can be produced from PAHs formed in the first cycle.

EXAMPLE 1

Heavy fuel oil was heated to 160°C and introduced in the reactor by use of the self-invented axial aligned nozzle at a feed rate of 67 kg per hour. The reactor pressure was kept at 2 bar. Hydrogen was employed as plasma gas, and the plasma gas feed rate was 350Nm³/h, while the gross power supply from the plasma generator was 620 kW. This resulted in a plasma gas enthalpy of 1.8 kWh/Nm³ H₂. The time elapsed from the atomised oil was introduced until the product left the reactor was approximately 0.23 seconds.

The resulting carbon black was traditional amorphous of N-7xx quality. The volatile content of the carbon black was measured to 0.6%.

EXAMPLE 2

In this example the oil feed rate, the hydrogen plasma gas enthalpy as well as the residence time was tuned in such a direction that the evaporated hydrocarbons did not achieve pyrolysis temperature during the first cycle. The residence time of the hydrocarbons during the first cycle through the reactor was minimised by increasing the oil and plasma gas feed rate.

Heavy fuel oil was heated to 160°C and introduced in the reactor by use of the self-invented axial aligned nozzle at a feed rate of 115 kg per hour. The reactor pressure was kept at 2 bar. The hydrogen plasma gas feed rate was 450Nm³/h, while the gross power of supply from the plasma generator was 1005 kW. This resulted in plasma gas enthalpy of 2.2 kWh/Nm³ H₂. The time elapsed from the oil was introduced until the PAHs left the reactor was approximately 0.16 seconds.

The resulting PAHs were reintroduced into the reactor in the plasma-arc zone to produce a micro-domain graphitic material, with a yield higher than 90%. The volatile content of the carbon material was measured to 0.7%. All other
5 process parameters were the same as for the first cycle.

Although in the example of the production of the hydrogen storage media has been described as a conversion of heavy oil to a micro-domain graphitic material, it should be understood that the media can be produced from
10 conversion of all hydrocarbons, both liquid and gaseous. Also, the production may be performed as a batch or continuous production, with one or more plasma reactors in series etc. Concerning micro-domain graphitic products where the PAHs formed in the first decomposition step is reintroduced into the same plasma reactor, the micro-domain graphitic materials formed in the
15 second decomposition step are of course separated from the PAH containing plasma gas by any conventional suited means. This may be by filtering, cyclones etc.

Further, any gas that is inert and do not pollute the micro-domain products may be used as plasma gas, but hydrogen is specially suited since it is a
20 product of the process. The plasma gas may be recycled back into the reactor, if desired. It is also possible to employ the present method by introducing additional hydrocarbons through inlets at the sides of the decomposition reactor to control the temperature in the decomposition zone and/or to
25 increase the yield, see the applicant's European patent EP 0 636 162.

REFERENCES

1. D. Huffman, Physics Today, p. 22, 1991.
2. T.W. Ebbesen, Physics Today, p. 26, 1996.
- 5 3. M. Ge and K. Sattler, Chemical Physics Letters 220, p. 192, 1994.
4. P. Li and K. Sattler, Mat. Res. Soc. Symp. Proc. 359, p. 87, 1995.
5. R. Vincent, N. Burton, P.M. Lister and J.D. Wright, Inst. Phys, Conf. Ser.,
138, p. 83, 1993.
6. Hydrogen & Fuel Cell Letter, vol. 7/No. 2, Feb. 1997.
- 10 7. R.M. Baum, Chem. Eng. News, 22, p. 8, 1993.
8. Japanese Patent JP 27801 A2, Fullerene- based hydrogen storage media,
18th August 1994.
9. A. Hirsch, Chemistry of Fullerenes, Thieme Verlag, Stuttgart, Ch. 5, p.
117, 1994.
- 15 10. B.P. Tarasov, V.N. Fokin, A.P. Moravsky, Y.M. Shul'ga, V.A. Yartys,
Journal of Alloys and Compounds 153-254, p. 25, 1997.
11. R.E Haufler, Y. Chai, L.P.F.m, Chibante, J. Conceico, C. Jin, L-S Wang,
S. Maruyama, R.E. Smalley, Mat. Res. Soc. Symp. Proc. 206, p. 627, 1991
- 20 12. M. Endo and H.W. Kroto, J. Phys. Chem. 96, p. 6941, 1992.

WHAT IS CLAIMED

1. A carbon media for storage of hydrogen,
c h a r a c t e r i s e d i n that it comprises a micro-domain graphitic
materials which have been produced by decomposition of hydrocarbons in a
5 reaction chamber connected to a plasma generator in which the hydrocarbons
are subjected to a first decomposition step, where the hydrocarbon is fed into
the decomposition chamber in the vicinity of the plasma arc zone and mixed
with the plasma gas, and where the process parameters are adjusted in such a
manner that the hydrocarbons do not reach pyrolysis temperature and are
10 only partially decomposed to form polycyclic aromatic hydrocarbons
(PAHs),
- that the hydrocarbons in the form of PAHs are, after the first
decomposition step, mixed with a plasma gas and reintroduced as a part of a
plasma gas into a plasma arc zone in a decomposition chamber and subjected
15 to a second decomposition step, where the intense heat in the plasma arc zone
causes the PAHs to be converted into the micro-domain graphitic materials.
2. A media according to claim 1,
c h a r a c t e r i s e d i n that the micro-domain graphitic materials consists
of at least one of the materials chosen from the group comprising carbon
20 nanotubes, fullerenes, carbon micro-cones, and flat graphitic carbon sheets.
3. A media according to claim 2,
c h a r a c t e r i s e d i n that the domain size is smaller than 5 μm in
diameter or length parallel to the graphitic stacking direction and having a
thickness of less than 100 nm in the graphitic stacking direction.
- 25 4. A media according to claim 1 - 3,
c h a r a c t e r i s e d i n that the media contains micro-domain graphitic
materials in the range from 0 to above 90wt%.
5. A media according to claim 4,
c h a r a c t e r i s e d i n that the media contains more than 90wt% micro-
30 domain graphitic materials.

6. A media according to any one of claims 1-5,
characterised in that the media results from dehydrogenation of
heavy fuel oil into micro-domain graphitic materials.
- 5 7. A carbon media for storage of hydrogen comprising micro-domain
graphitic materials,
characterised in that it contains open carbon micro-cones with total
disclination degrees 60° and/or 120° , corresponding to cone angles of
respectively 112.9° and/or 83.6° .
- 10 8. A carbon media for storage of hydrogen,
characterised in that it comprises a micro-domain material which
have been produced by decomposition of hydrocarbons in a reaction chamber
connected to a plasma generator in which the intense heat in the generator
causes the hydrocarbons to be converted into the micro-domain material.
- 15 9. A media according to claim 8,
characterised in that the micro-domain material comprises
amorphous carbon such as conventional carbon black.
- 20 10. A media for storage of hydrogen,
characterised in that the micro-domain material comprises a
mixture of the graphitic micro-domain materials given claims 1-7 micro-
domain materials given in claims 8-9.
11. Use of the carbon media as given in claims 1-10 for storage of hydrogen.

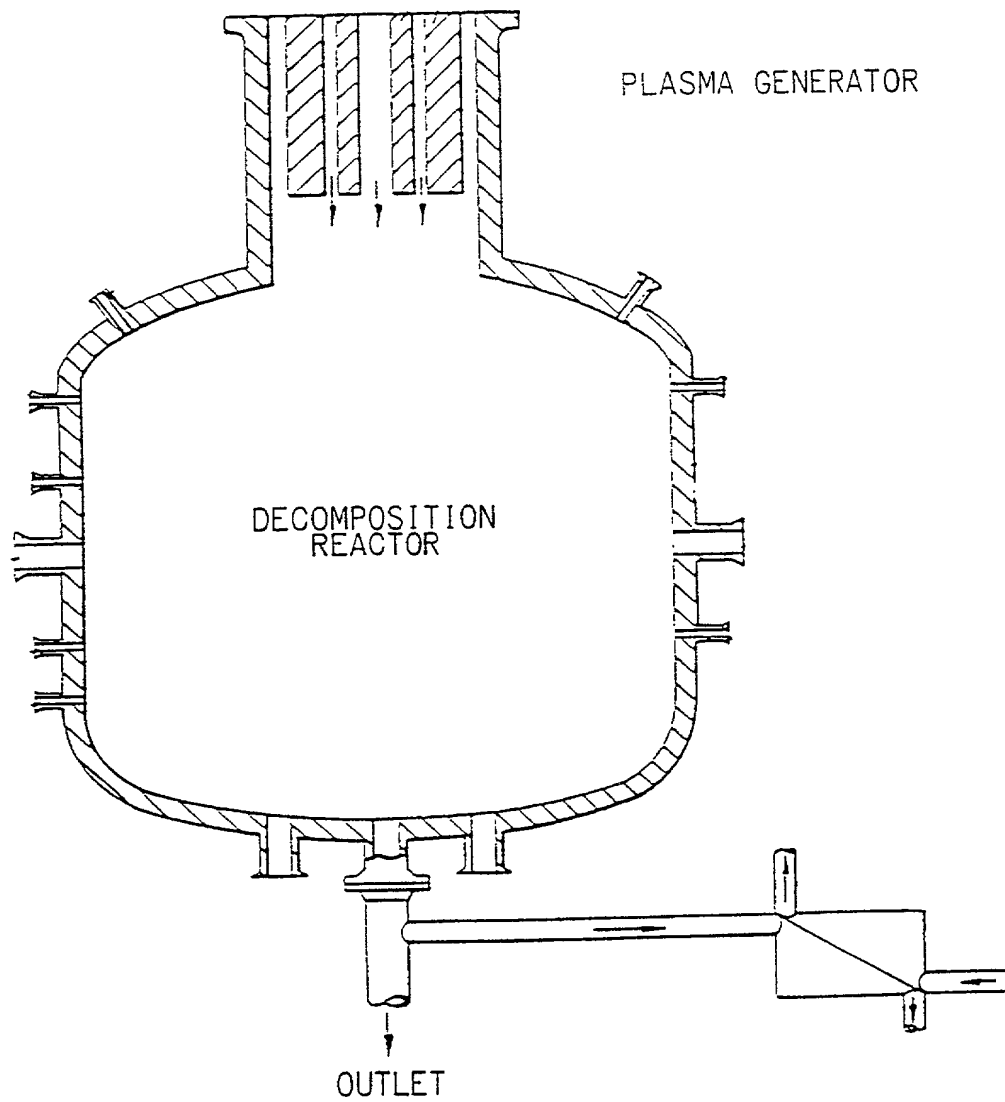


FIG. 1

Total Disclination

Projected Angles

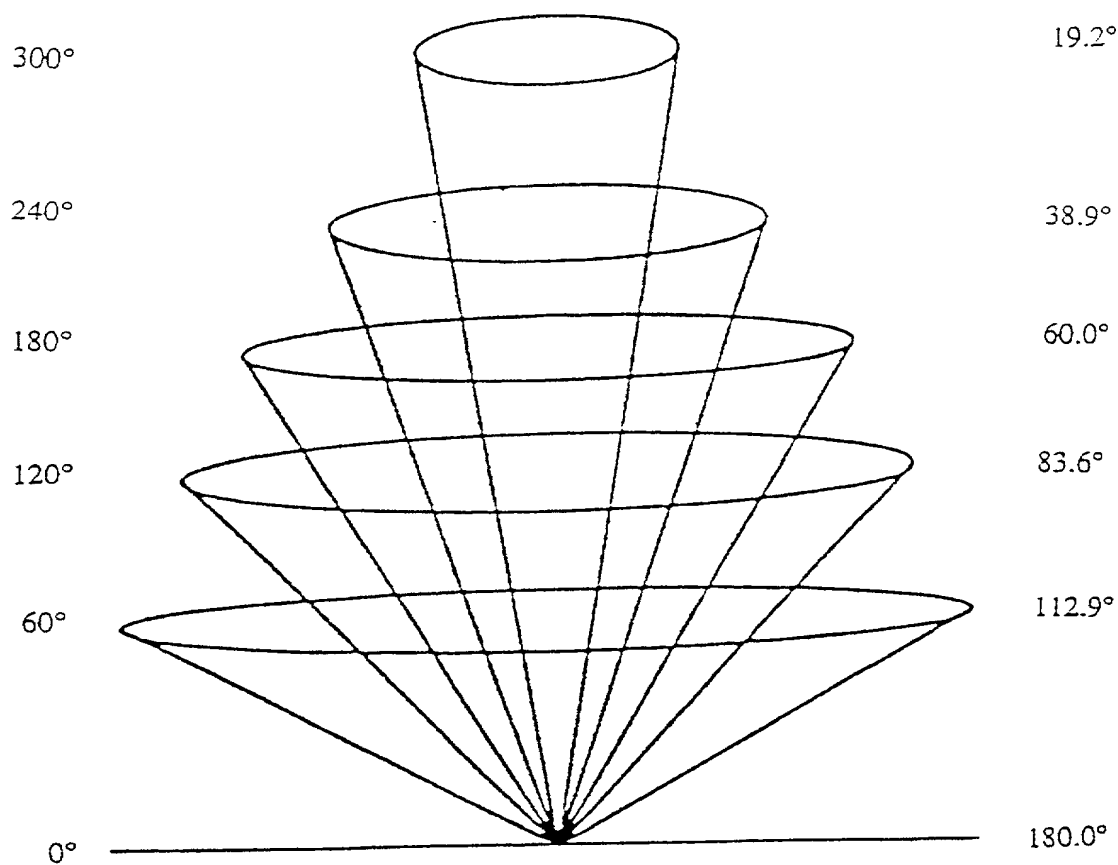


Fig. 3

4/8

09762211

50mm

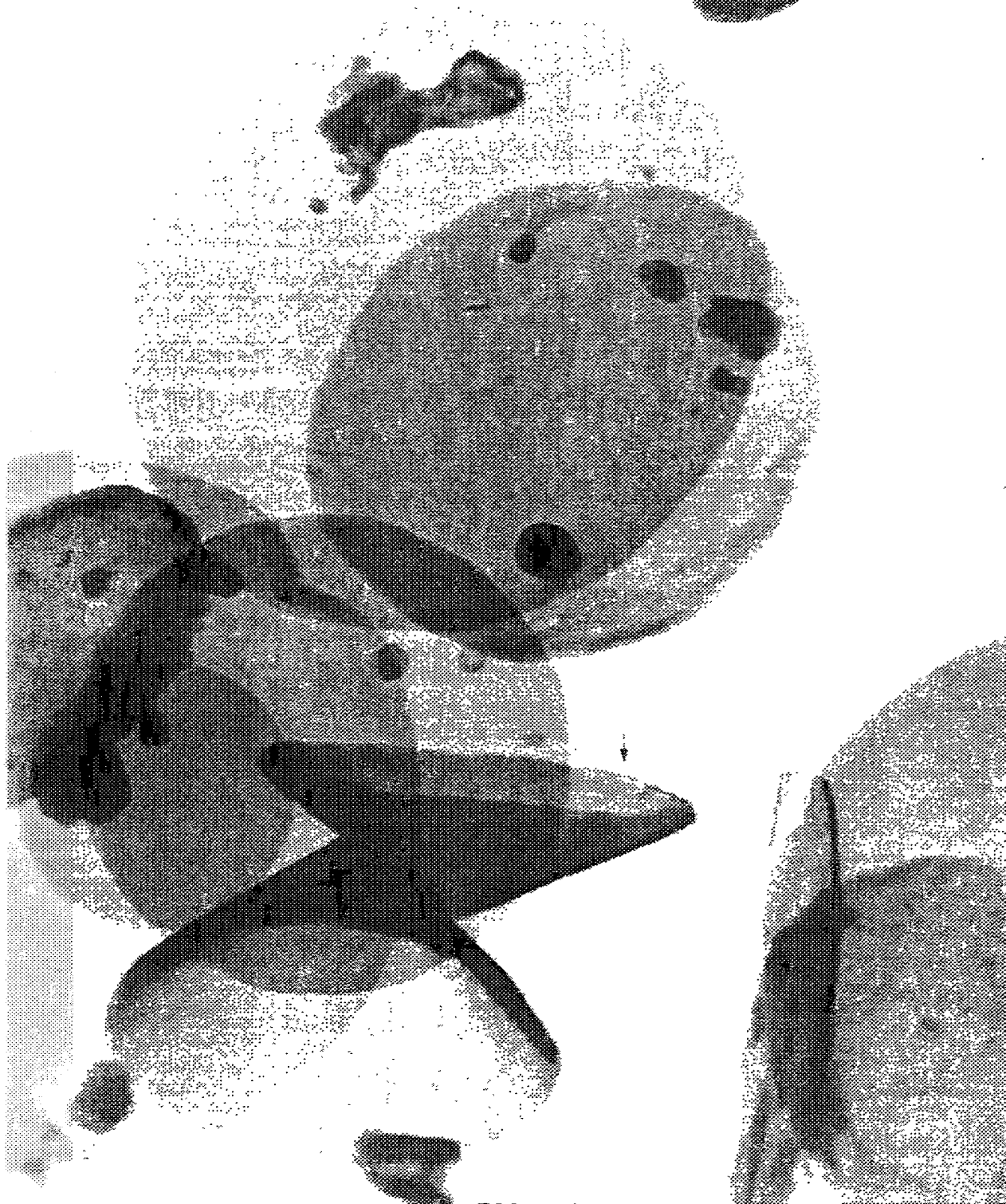


FIG. 4A



FIG. 4B

09762211

50nm

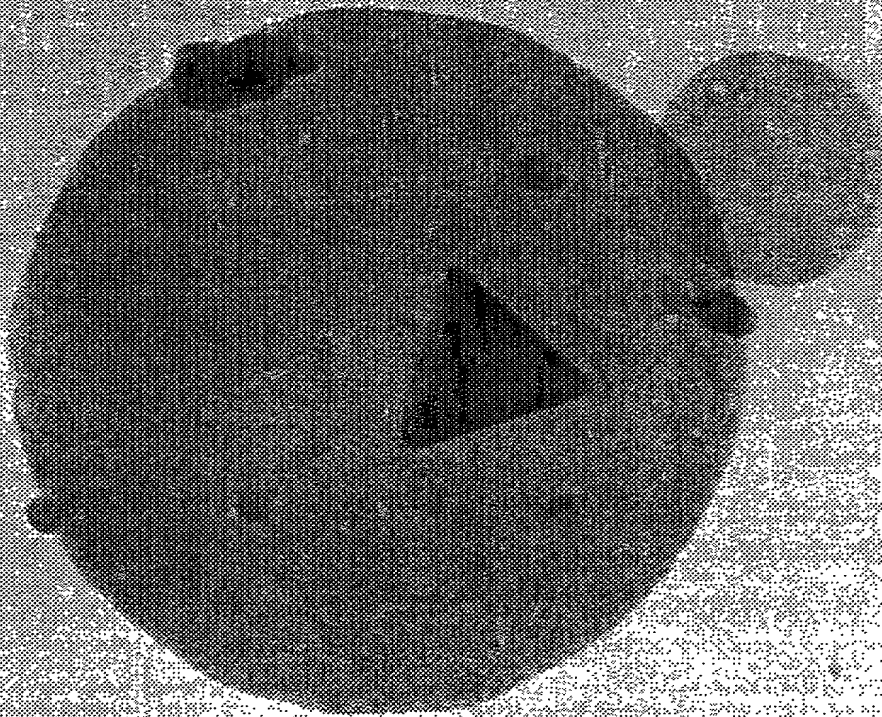


FIG. 4C

50mm

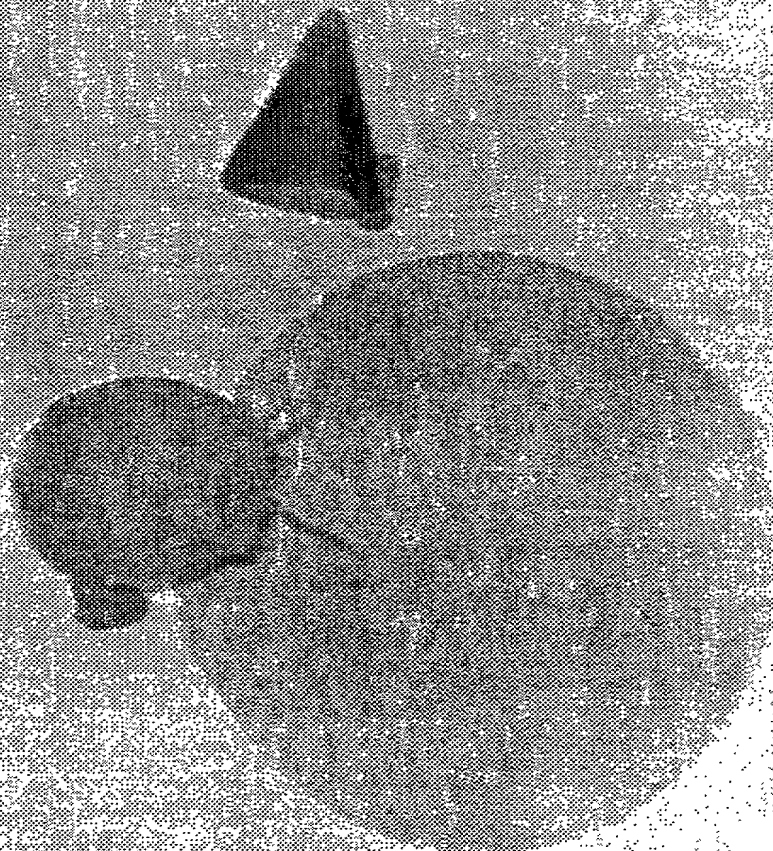


FIG. 4D

3/8

09762211

50mm

09762211 09762211

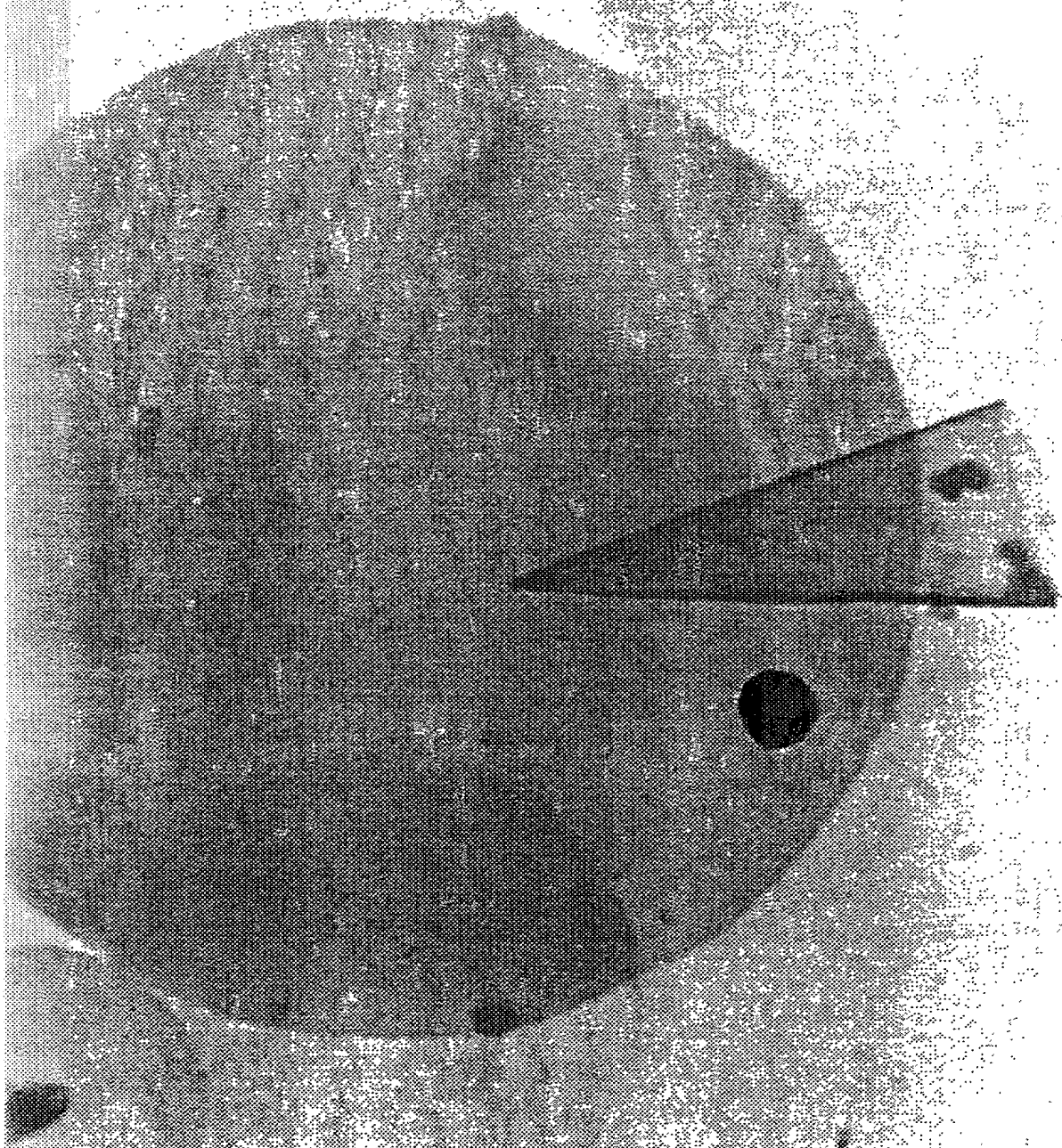


FIG. 4E

RULE 63 (37 C.F.R. 1.63)
DECLARATION AND POWER OF ATTORNEY FOR PATENT APPLICATION IN THE
UNITED STATES PATENT AND TRADEMARK OFFICE

As a below named inventor, I hereby declare that my residence, post office address and citizenship are as stated below next to my name, and I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the INVENTION ENTITLED
Carbon media for storage of hydrogen the specification of which is attached hereto.

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above. I acknowledge the duty to disclose all information known to me to be material to patentability as defined in 37 C.F.R. 1.56. I hereby claim foreign priority benefits under 35 U.S.C. 119/365 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate filed by me or my assignee disclosing the subject matter claimed in this application and having a filing date (1) before that of the application on which priority is claimed, or (2) if no priority claimed, before the filing date of this application:

PRIOR FOREIGN APPLICATION(S)			Date first Laid- open or Published	Date Patented or Granted	Priority Claimed Yes No
Number	Country	Day/MONTH/Year Filed			
19984497	Norway	25/09/1998			X

I hereby claim domestic priority benefit under 35 U.S.C. 120/365 of the indicated United States applications listed below and PCT international applications listed above or below and, if this is a continuation-in-part (CIP) application, insofar as the subject matter disclosed and claimed in this application is in addition to that disclosed in such prior applications, I acknowledge the duty to disclose all information known to me to be material to patentability as defined in 37 C.F.R. 1.56 which became available between the filing date of each such prior application and the national or PCT international filing date of this application:

PRIOR U.S. PROVISIONAL, NONPROVISIONAL AND/OR PCT APPLICATION(S)		Status	Priority Claimed
Application No. (series code/serial no.)	Day/MONTH/Year Filed	pending, abandoned, patented	Yes No
PCT/NO99/00287	17/09/1999		X

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

I hereby appoint the registered practitioners represented by Customer No.: 20736 to prosecute this application and transact all business in the U.S. Patent and Trademark Office in connection therewith. Direct all correspondence to Farkas & Manelli, PLLC at Customer No.: 20736.

1. INVENTOR'S SIGNATURE: Steinar Lynum Date 30.01.01

1-00 Inventor's Name (typed) Steinar Lynum Norway
First Middle Initial Family Name Country of Citizenship
Residence (City) Oslo
Post Office Address (Include Zip Code) Hartmannsvei 40b, N-0284 OSLO NORWAY

2. INVENTOR'S SIGNATURE: Jan Huggdahl Date 30.01.01

2-00 Inventor's Name (typed) Jan Huggdahl Norway
First Middle Initial Family Name Country of Citizenship
Residence (City) Trondheim
Post Office Address (Include Zip Code) Harald Bothners vei 10, N-7051 TRONDHEIM NORWAY

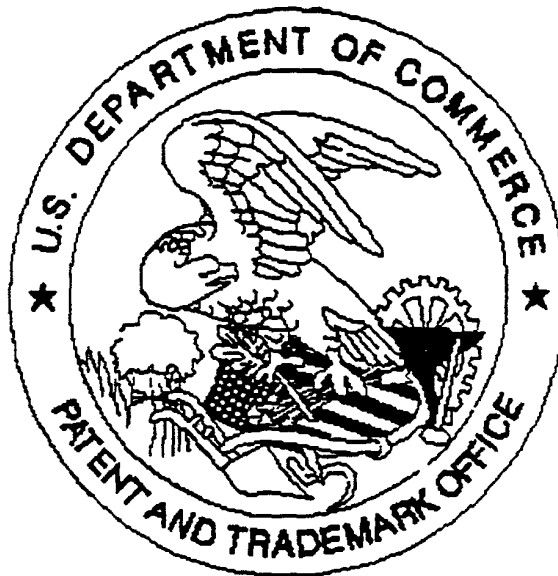
3. INVENTOR'S SIGNATURE: Ketil Hox Date 30.01.01

3-00 Inventor's Name (typed) Ketil Hox Norway
First Middle Initial Family Name Country of Citizenship
Residence (City) Trondheim
Post Office Address (Include Zip Code) Gjørtlerveien 10, N-7021 TRONDHEIM NORWAY

4. INVENTOR'S SIGNATURE: Ragne Hildrum Date 31.01.01

4-00 Inventor's Name (typed) Ragne Hildrum Norway
First Middle Initial Family Name Country of Citizenship
Residence (City) Oslo
Post Office Address (Include Zip Code) Nilsrudkleiva 11, N-0874 OSLO NORWAY

United States Patent & Trademark Office
Office of Initial Patent Examination – Scanning Division



Application deficiencies found during scanning:

☐ Page(s) _____ of _____ were not present
for scanning. (Document title)

☐ Page(s) _____ of _____ were not present
for scanning. (Document title)

☒ *Scanned copy is best available.*
DRAWINGS